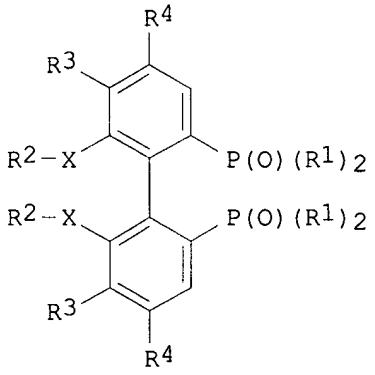
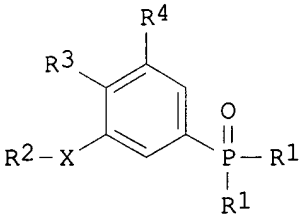
L3 ANSWER 165 OF 212 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2000:37891 CAPLUS <<LOGINID::20070401>>
DOCUMENT NUMBER: 132:93468
TITLE: Preparation of biphenyl diphosphine oxide by lithiation and oxidative coupling of phenylphosphine oxide
INVENTOR(S): Yokozawa, Susumu; Saito, Takao; Sayo, Noboru; Ishizaki, Takeo
PATENT ASSIGNEE(S): Takasago Perfumery Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 14 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000016997	A	20000118	JP 1998-181027	19980626
JP 3146187	B2	20010312		
PRIORITY APPLN. INFO.:			JP 1998-181027	19980626

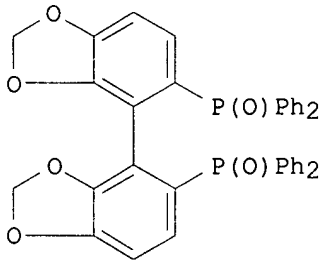
OTHER SOURCE(S): CASREACT 132:93468; MARPAT 132:93468
GI



I



II



III

AB The title compds. [I; R1 = cycloalkyl, (un)substituted Ph, naphthyl, pyridyl, quinolyl, isoquinolyl, furfuryl, benzofurfuryl, thienyl, or benzothienyl; R2 = lower alkyl, lower ether, lower haloalkyl, Ph; X = hetero atom; R3, R4 = hydrogen, halogen, lower alkyl, lower alkoxy, di(lower alkyl)amino, lower haloalkyl, Ph; or R2 and R2 or R3 and R4 are linked to each other to form a ring] are prepared by treatment of phosphine oxide (II; R1 - R4, X = same as above) with base followed by dimerization using oxidizing agent. I are useful as intermediates for diphosphine

compds. which are ligands of metal coordination compds. for an synthesis catalyst. Thus, a solution of 75.22 g diphenyl(3,4-methylenedioxyphenyl)phosphine oxide in 300 mL THF was added dropwise at -10° to -5° to a solution of lithium diisopropylamide prepared by treatment of 40 mL diisopropylamine in THF with 175 mL 1.7 M BuLi solution and stirred at -12° for 15 min to give a lithium reagent which was added to 5.79 g FeCl3 in 150 mL toluene and 150 mL THF under ice-cooling at 8-10° over 30 min and stirred at room temperature overnight to give 74.8% biphenyl bisphosphine oxide (III).

IT 133545-15-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of biphenyl diphosphine oxide by lithiation and oxidative coupling of phenylphosphine oxide)

RN 133545-15-0 CAPLUS

CN Phosphine oxide, (6,6'-dimethoxy[1,1'-biphenyl]-2,2'-diyl)bis[diphenyl-(9CI) (CA INDEX NAME)